3.—A GRAZING INCIDENCE METHOD FOR THE DETERMINATION OF HIGH REFRACTIVE INDICES.

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The determination of the refractive indices of minerals is usually done indirectly. The refractive index of a liquid or fusible solid medium is made to agree with that of the mineral and the index of the medium is then determined.

Fragments of the mineral are immersed in the medium and their refractive indices compared, using a polarizing microscope. The comparison is effected by well-known Becke Line or shadow methods, or by means of special methods such as that described by Saylor (1935). Suitably oriented grains are selected by optical methods (Slawson and Peck, 1936) or else grains are made to lie in the desired position by use of a universal stage. The refractive index of the medium is commonly changed by mixing two selected media, one with a higher refractive index than the mineral and the other a lower index: the proportions are varied until the index of the mixture matches that of the mineral grain. The refractive index of the immersion medium may be varied by other means; the wave-length of light (Posnjak and Merwin, 1922) or the temperature (Gaubert, 1922), or both the wave-length of light and the temperature may be varied (Winchell and Emmons, 1926). Whatever method is adopted for matching the refractive index of the immersion medium to that of the mineral, there still remains the determination of the refractive index of the medium at the temperature and for the wave-length of light used.

A search of mineralogical literature has shown that the refractive indices of immersion media of high refractive index are almost universally determined on a goniometer by Fraunhofer's method of minimum deviation. A hollow prism is used for liquids and solid media are moulded between cover-slips to form prisms.

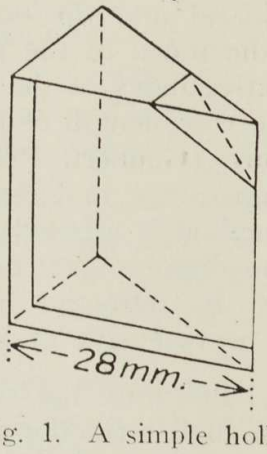
Of the several methods available for determining refractive indices with a prism, there are two that are especially suitable for use with the goniometer, namely, Fraunhofer's method of minimum deviation already mentioned and Kohlrausch's method of critical or grazing incidence. It appears that the latter method has been overlooked entirely by mineralogists: it offers certain advantages of speed and convenience over that commonly employed, and an accuracy satisfactory for most mineralogical purposes is easily obtained with the simplest accessories on any goniometer which can be read to one minute of arc.

An advantage that may appeal to some is that the instrument can be used without disturbing it from its adjustment for the examination

of crystals. Another point of appeal is that, unlike the determination of the position of minimum deviation, only one reading of each position of the prism is necessary, these positions being ascertained with ease and precision.

While a sodium lamp or flame is desirable and indeed seems necessary in the high ranges of solid media, values which are quite satisfactory for purposes of identification can be obtained for liquid media using an ordinary white light, making all adjustments to the yellow-green portion of the spectrum.

The hollow prism used by the author has a refracting angle of fifty degrees, while another of forty degrees is also available. These are made after the pattern used by Ross, described by Larsen (1921): the solid prism has one corner of the refracting edge bevelled off to form the hollow prism when two one-inch square portions of good quality microscope slide are cemented to the solid prism with deKhotinsky's cement, as shown in text fig. 1. No attempt was made to select the glass by interference methods, checking for parallelism of perfectly flat surfaces; the only check made was to observe the undistorted reflection of a straight edge of a window, first from one surface and then the other. For other than room temperatures, a prism similar to that described by Butler (1933) can be used.



Text fig. 1. A simple hollow prism.

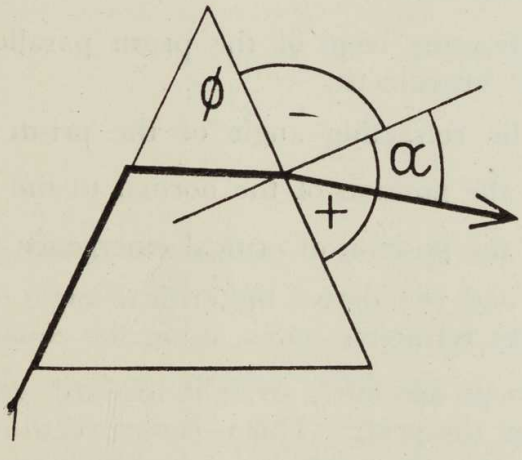
Hollow prisms of such simple pattern have been found to be quite stable and may be sluiced repeatedly with alcohol and dried in a blast of cold air without altering the refracting angle; consequently a graph can be drawn for each prism used, saving much of the time taken in calculating the refractive indices from the data obtained.

Prisms of solid media are made by melting the medium into the angle between two pieces of thin microscope slide; number one coverslips were found to be too thin, as they distort too readily as the melt solidifies. The other chief difficulty in obtaining prisms of solid media, that of obtaining a clear rather than a frosted refracting edge, is best overcome by being sufficiently liberal in the use of the medium.

In the intermediate range of refractive indices, from 1·55 to 1·67, indices determined by this method with such simple accessories as

described were compared with those obtained on the same liquids using an Abbe refractometer made by Hilger: they were found to differ by not more than 0·0002. Lacking any means of direct comparison for the high ranges, a series of determinations was run on thallium bromide, one of the solid media to which attention has been drawn by Barth (1929): the average was 2·425 and the maximum deviation therefrom was 0·006. Barth gives 2·42 as the refractive index of this medium.

The principle of the method is illustrated in text fig. 2. Light from a sodium-lamp impinges at grazing incidence upon the incident face of the prism and is refracted into the prism and again refracted upon passing through the emergent face. The angle between the direction of emergence of the light and the normal to the emergent face of the prism is measured; this angle, and the refracting angle of the prism, constitute all the data necessary for the calculation of the refractive index.



Text fig. 2. The path of light at grazing incidence.

The equation relating the refractive index of the prism to the refracting angle and the angle of emergence, that is, the angle between the emergent light beam and the normal to the emergent face, can be cast into the form

$$n = \sqrt{\left(\frac{\cos \phi + \sin \alpha}{\sin \phi}\right)^2 + 1}$$

where n = refractive index of the prism,

ϕ = refracting angle of the prism,

α = critical angle of emergence, that is, the angle between the normal to the emergent face and the position of emergence at grazing incidence.

The critical angle of emergence, α, is positive if in the quadrant between the normal and the base, as shown in text fig 2, and negative if between the normal and the refracting angle.

For those who use the popular Goldschmidt two-circle goniometer, an outline of the method as developed by the author at the Government Chemical Laboratories, Perth, for application to the instrument as made by Stoe & Cie, m.b.h., is given here. Suitable modifications are readily made to apply the method to other types and to single-circle goniometers.

Assuming the instrument to be in adjustment, the steps in the determination are as follow:—

(1) Set up the prism in the goniometer so that the refracting edge is parallel to one of the plane slides and vertical, and so placed that the plane of the incident face clears the vertical circle (the horizontal-axis graduated circle).

(2) Set the prism so that both the vertical axis of the instrument and the telescope axis pass through it, using the carriage slide and the plane slides.

(3) Set the refracting edge of the prism parallel to the vertical axis of the instrument.

(4) Measure the refracting angle of the prism.

(5) Determine the position of the normal to the emergent face.

(6) Determine the position of critical emergence.

(7) From (5) and (6) derive the critical angle of emergence and calculate the refractive index, using the equation given above.

The first two steps are fairly straight forward, requiring but little ingenuity in mounting the prism. This is cemented to its supporting peg or pin in such a way that the prism is perched high above the horizontal axis of the peg; the incident face of the prism then comes above the level of the horizontal rocking screw arc which otherwise would cast a shadow on it. The plane of the incident face is made to clear the vertical graduated circle by means of the horizontal rocking screw.

Having set the telescope lenses to give an enlarged image of the target, which combination is used throughout the determination, the refracting edge is made parallel to the vertical axis in the usual way so that, no matter which face of the prism reflects the target, its image remains on the horizontal cross-hair.

If a series of refractive indices is being determined, the prism may be re-set accurately in its holder after each cleaning by first setting it vertical, as near as can be, by eye, and then turning it about the vertical axis to bring the reflection of the target into the field of the telescope; if off-centre, loosen the clamp slightly, turn the prism about its supporting peg until the image is centred and re-clamp firmly. If carefully done, much time can be saved, for the prism can be set in the same

position every time: it becomes necessary then only quickly to check the setting and the position of the normal to the emergent face.

To determine the position of the normal to the emergent face, the same arrangement is used as for adjusting the telescope by auto-collimation. The prism is swung until the image of the cross-hairs reflected from the emergent face of the prism coincides with the direct image. In this position the axis of the telescope is normal to the emergent face; the horizontal circle is read and noted as the position of the normal.

The sodium lamp is placed at the same distance as the target from the vertical axis and at the same height and in such a position that the light from it reaches the incident face of the prism at a moderate angle, some ten to thirty degrees or so, from the direction of the base of the prism. The prism is swung about the vertical axis until a sharp image of the lamp is seen in the telescope. Retaining the image of the lamp in the field and keeping the lamp at the same distance from the vertical axis, move the lamp towards the plane of the incident face, swinging the prism as necessary. This causes the image of the lamp to become attenuated, so much so that finally it becomes the merest streak of light. This streak is placed behind the vertical cross-hair. The horizontal circle is read, noting this as the position of critical emergence.

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